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## EXPERIMENTAL UNCERTAINTY IN LASER-BASED OPTICAL DENSITY MEASUREMENTS

by

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are derived, which can be used t	o generate a unique tolerance f	for each experimental option	cal density measurement performed.
The laboratory setup is described	d and a sample experimental ru	in is presented, which com	npares ODs obtained using a UV/vis
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#### TABLE OF CONTENTS

LIST OF FIGURES	iv
PREFACE	v
INTRODUCTION	1
EXPERIMENTAL SETUP	1
EXPERIMENTAL PROCEDURE	2
OPTICAL DENSITY CALCULATIONS	3
EXPERIMENTAL UNCERTAINTIES	3
ESTIMATE OF ABSOLUTE TOLERANCES	4
DATA ANALYSIS	5
SAMPLE EXPERIMENTAL DATA RUN	6
CONCLUSIONS	7
REFERENCES	8
DISTRIBUTION LIST	9

## LIST OF FIGURES

1.	Experimental setup for optical density measurements.	2
2.	Example of Table showing data from actual experimental run.	6

#### PREFACE

This report summarizes the development and application of an uncertainty analysis used to define the tolerances in experimentally determined optical density values of laser protective materials and devices. This work is being done under the project <u>Laser Eye Protection</u>, 62786A/AH98/CA, administered by the Directed Energy Team, Multifunctional Materials Group, Survivability Directorate, Natick Research, Development, and Engineering Center, U.S. Army Soldier Systems Command. The citation of trade names in this report does not constitute official endorsement or approval of use of an item.

## EPERIMENTAL UNCERTAINTY IN LASER-BASED OPTICAL DENSITY MEASUREMENTS

#### INTRODUCTION

In order to evaluate eye-protective systems against laser threats, researchers need to determine the degree to which such systems attenuate incident laser radiation. This energy attenuation is generally stated in terms of optical density (OD) which is defined as follows:

$$OD = Log_{10} (1/T), (1)$$

where T is the measured transmission (output/input). For these purposes optical density experimental setup description and measurement procedures are documented in the Performance Specifications for the Ballistic/Laser Protective Spectacle (BLPS)<sup>1</sup> and the Special Protective Eyewear, Cylindrical System (SPECS)<sup>2</sup>. Laser wavelengths and pulse widths vary with the optical system being tested<sup>3</sup>. In general, measurements are performed at 532, 694 and 1064 nm. For pulse widths greater than 1 ns and less than 40 ns, specifications require a fluence of 20 mJ/cm<sup>2</sup>.

When reporting experimentally determined optical density values of a system it is necessary for investigators to give an associated tolerance. This report describes the current method being used to obtain OD values and the derivation and implementation of uncertainty expressions used to generate the tolerances. A sample experimental run is presented in which three lenses are evaluated. Results show that the experimentally determined OD values remain within predicted tolerances.

#### EXPERIMENTAL SETUP

To ensure uniformity of local fluences, it is necessary to produce a flat spatial profile, either by expanding the gaussian laser beam profile with a beam expander or through the natural divergence of the beam and then removing all but the relatively flat center portion of the beam with a 5 mm aperture (Figure 1). Approximately 10% of the beam energy is diverted at the beam sampler to a reference detector used to monitor, and correct for, fluctuations in beam energy. The remaining energy passes through the 5 mm diameter pin hole and is incident on the test sample. The transmitted energy is measured at the throughput detector. Neutral density (ND) filters are used to attenuate beam energy to below the damage threshold of the detectors with

corrections made during data analysis.

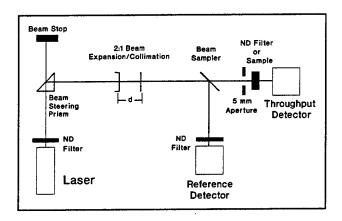


Figure 1. Experimental setup for optical density measurements

#### EXPERIMENTAL PROCEDURE

Optical density (OD) measurements are made by first recording a baseline. This consists of recording energy measurements with a calibrated ND filter in the throughput path. All subsequent energy measurements are then compared to these readings. This technique assumes a negligible change in laser output through the course of the experimental run.

Energy measurements are then recorded for a set of calibrated ND filters for which the least and greatest optical densities depend on the dynamic range of the throughput detector. In general, the filters range from an OD of about 1 or 2, to an OD of about 5. The purpose of the calibration run is to demonstrate that the system is set up and operating properly and to show the experimental uncertainties at intervals over the dynamic range of the system. The OD filters are pre-calibrated with a UV/vis spectrophotometer at the wavelengths of interest. For all energy measurements, 25 shots are averaged.

Immediately following the calibration run, the test samples are evaluated in the same fashion. One more run is then made with a single calibrated ND filter to check that the laser output power has remained relatively constant over the time required to complete the run. At each wavelength the beam is blocked and a noise data file is recorded for later subtraction from energy measurements.

#### OPTICAL DENSITY CALCULATIONS

Optical densities are calculated using the following expression:

$$OD = D - F + Log \left[ \frac{E_m}{E_x} * \frac{E_x'}{E_m'} \right], \tag{2}$$

where D is the OD of the throughput ND filter used in recording the baseline, F is the OD of the ND filter placed in front of the throughput detector during data acquisition to prevent damage to the detector if the sample OD is too low,  $E_{\rm m}$  is the baseline throughput energy,  $E_{\rm r}$  is the baseline reference energy,  $E'_{\rm m}$  is the test-sample throughput energy and  $E'_{\rm r}$  is the test-sample reference energy.

#### EXPERIMENTAL UNCERTAINTIES

The uncertainty analysis is based on the standard root-sum-square method. The derivation of the uncertainty equations is outlined below.

If it is assumed that the uncertainties  $(W_1,\ W_2,\ldots,\ W_n)$  in the independent variables  $(X_1,\ X_2,\ldots,\ X_n)$  are all given with the same probability, then the uncertainty  $(W_R)$  in the dependent variable (R) is given by the expression

$$W_{R} = \left[ \left( \frac{\partial R}{\partial X_{1}} W_{1} \right)^{2} + \left( \frac{\partial R}{\partial X_{2}} W_{2} \right)^{2} + \ldots + \left( \frac{\partial R}{\partial X_{n}} W_{n} \right)^{2} \right]^{1/2}. \tag{3}$$

By inserting Eq. 2 into Eq. 3, a probable estimate of the uncertainty in experimentally determined OD values can be obtained:

$$W_{OD} = \left[ \left( \frac{\partial OD}{\partial D} W_D \right)^2 + \left( \frac{\partial OD}{\partial F} W_F \right)^2 + \left( \frac{\partial OD}{\partial E_m} W_{E_m} \right)^2 + \left( \frac{\partial OD}{\partial E_r} W_{E_r} \right)^2 + \left( \frac{\partial OD}{\partial E_r'} W_{E_r'} \right)^2 + \left( \frac{\partial OD}{\partial E_m'} W_{E_m'} \right)^2 \right]^{1/2},$$

$$(4)$$

where

$$\frac{\partial OD}{\partial D} = 1, \tag{5}$$

$$\frac{\partial OD}{\partial F} = -1, \tag{6}$$

$$\frac{\partial OD}{\partial E_m} = \frac{1}{E_m \ln 10},\tag{7}$$

$$\frac{\partial OD}{\partial E_r} = \frac{-1}{E_r \ln 10},\tag{8}$$

$$\frac{\partial OD}{\partial E_r'} = \frac{1}{E_r' \ln 10} \tag{9}$$

and

$$\frac{\partial OD}{\partial E'_m} = \frac{-1}{E'_m \ln 10}.$$
 (10)

#### ESTIMATE OF ABSOLUTE TOLERANCES

Experimental tolerances are calculated for each OD measurement to ensure the integrity of the experimental run. In order to evaluate Eq. 4, it is necessary to obtain estimates for the uncertainties  $W_{\rm D}$ ,  $W_{\rm F}$ ,  $W_{\rm Em}$ ,  $W_{\rm Er}$ ,  $W_{\rm E,m}$  and  $W_{\rm F,r}$ .

A UV/vis/near-infrared Perkin-Elmer Lambda 9 spectrophotometer was used to determine OD values of the ND filters D and F. The manufacturer-stated tolerance for these readings is  $\pm$  0.08%. A percent uncertainty of 0.1% is used in the data analysis software to determine  $W_{\rm D}$  and  $W_{\rm F}$ .

Molectron J4-09 pyroelectric detectors are used with a Molectron JD2000 Joulemeter Ratiometer to obtain the energy measurements  $E_{\rm m}$ ,  $E_{\rm r}$ ,  $E'_{\rm m}$  and  $E'_{\rm r}$ . The detectors and joulemeter have been calibrated by the manufacturer using test equipment and standards traceable to the National Institute of Standards.

Manufacturer-stated tolerances for the J4-09 detectors are:

- Detector Linearity: 0.2%
- Calibration Uncertainty: ±5%

For the JD2000 Joulemeter Ratiometer:

- Joulemeter Linearity: 1% full scale
- Ratio Accuracy: 0.1%

The J4-09 calibration uncertainty of 5% was not considered as the absolute error in the energy terms  $E_{\rm m}$ ,  $E_{\rm r}$ ,  $E_{\rm m}'$  and  $E_{\rm r}'$  because Eq. 2 deals with energy ratios and not absolute values. Because of this, linearity is of more concern than the absolute calibrated uncertainty. Specifications for the J4-09 pyroelectric detector and power meter given above indicate only a slight divergence from linearity. These effects are minimized by recording the baseline energy measurements  $E_{\rm m}$  and  $E_{\rm r}$  with a ND filter of 2 or 3, thereby keeping the measurements near the midpoint of the dynamic range.

Of greater significance are the shot-to-shot fluctuations in laser output power. These vary with wavelength and other factors such as fluctuations in line source voltage and laser operating temperature. Standard deviations calculated over 25 shots show variations of about 2%. Shot-to-shot fluctuations are considered the primary source of uncertainty in energy measurements and are used in the analysis.

#### DATA ANALYSIS

Data are acquired in units of joules via the output of the joulemeter using manufacturer-supplied software. Data are imported into a commercial spreadsheet program for analysis. Absolute uncertainties  $W_{\rm D}$  and  $W_{\rm F}$  are calculated automatically when the values of D and F are entered at their respective locations in the spreadsheet. Baseline energy measurements for  $E_{\rm m}$  and  $E_{\rm F}$  are imported and averaged, noise is subtracted, and a standard deviation is calculated for the reference and throughput energy measurements. Baseline standard deviations ( $W_{\rm Em}$  and  $W_{\rm Er}$ ) are then entered at a specific location on the spreadsheet for use in subsequent uncertainty calculations.

Calibration and test sample energy measurements for  $E'_{m}$  and  $E'_{\Gamma}$  are imported and averaged, noise is subtracted, and standard deviations ( $W_{E'm}$  and  $W_{E'_{\Gamma}}$ ) are calculated. With these data, Eq. 2 automatically calculates the OD of the calibration and test samples and Eq. 4 calculates the associated uncertainty.

#### SAMPLE EXPERIMENTAL DATA RUN

Figure 2 shows the data table for an actual experimental run in which three lenses were evaluated at 532, 694 and 1064 nm. The top section of the table gives pertinent experimental parameters. The average fluences were determined by averaging the fluences over the 25 shots recorded for each datum and then averaging all the averages for the entire run for each wavelength.

WAVELENGTH	532 nm	694 nm	1064 nm
PULSE WIDTH	10 ns	30 ns	10 ns
REPETITION RATE	10 Hz	0.2 Hz	10 Hz
SPOT DIAMETER	5 mm	5 mm	5 mm
AVERAGE FLUENCE (per pulse)	20.3 mJ/cm <sup>2</sup>	21.9 mJ/cm <sup>2</sup>	21.0 mJ/cm <sup>2</sup>
CALIBRATION	OD (532 nm)	OD (694 nm)	OD (1064 nm)
SAMPLE	<u>exp</u> <u>std</u>	<u>exp</u> <u>std</u>	exp std
ND-1	Out of Range	Out of Range	1.10 1.10
ND-2	2.08 2.08	1.68 1.68	2.13 ±0.02 2.15
ND-3	3.11 ±0.02 3.10	2.48 ±0.05 2.44	2.49 ±0.02 2.50
ND-4	4.18 ±0.04 4.14	3.32 ±0.04 3.29	3.55 ±0.02 3.55
ND-5	Out of Range	4.19 ±0.05 4.14	4.62 ±0.04 4.61
ND-6	Out of Range	5.17 ±0.20 5.00	Out of Range
TEST SAMPLE	OD (532 nm)	OD (694 nm)	OD (1064 nm)
1	3.12 ±0.05	4.62 ±0.20	4.56 ±0.04
2	2.89 ±0.03	4.45 ±0.20	4.56 ±0.04
3	3.49 ±0.05	>5.00	3.47 ±0.02

Figure 2. Example of Table showing data from actual experimental run.

The middle section contains the results of the calibration run. At each wavelength the experimental OD is given in the exp column along with an associated uncertainty. The spectrophotometer data are the standard to which

the experimental ODs are compared. These values are given in the *std* column. The *Out of Range* entries in row ND-1 indicate throughput energies above the detector damage threshold at 532 and 694 nm. The *Out of Range* entries at the higher NDs indicate throughput energies that are too low to be reliably measured by the detectors. At each wavelength, the smallest experimental ODs are given without a tolerance. These are the OD values associated with the ND filters used to define the respective baselines. The ODs of these filters were determined with a spectrophotometer and are manually entered into the spreadsheet. Their tolerance is that of the spectrophotometer (±0.1%). Note that when the respective tolerance is taken into account, the laser-based OD measurements are consistent with those of the spectrophotometer in every case.

Finally, the last section in the table gives the results for the three test samples. The value of >5.00 given for Sample 3 at 694 nm indicates that the OD is outside the dynamic range of the system. This value represents the highest reliable OD evaluated in the calibration run.

#### CONCLUSIONS

The method currently being used to evaluate the OD of laser eye protection systems is outlined. The method conforms in all aspects to that described in relevant military performance specifications. Uncertainty expressions are derived, which are currently being used to generate a unique tolerance for each reported OD value. A sample experimental run is presented, which compares ODs obtained using a UV/vis spectrophotometer to those obtained with the described laser system. In all cases the ODs of the laser-based system fall within the predicted tolerances.

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  Requirements, 2 June 1997.

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